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Conversion of Municipal Sludge to Oil

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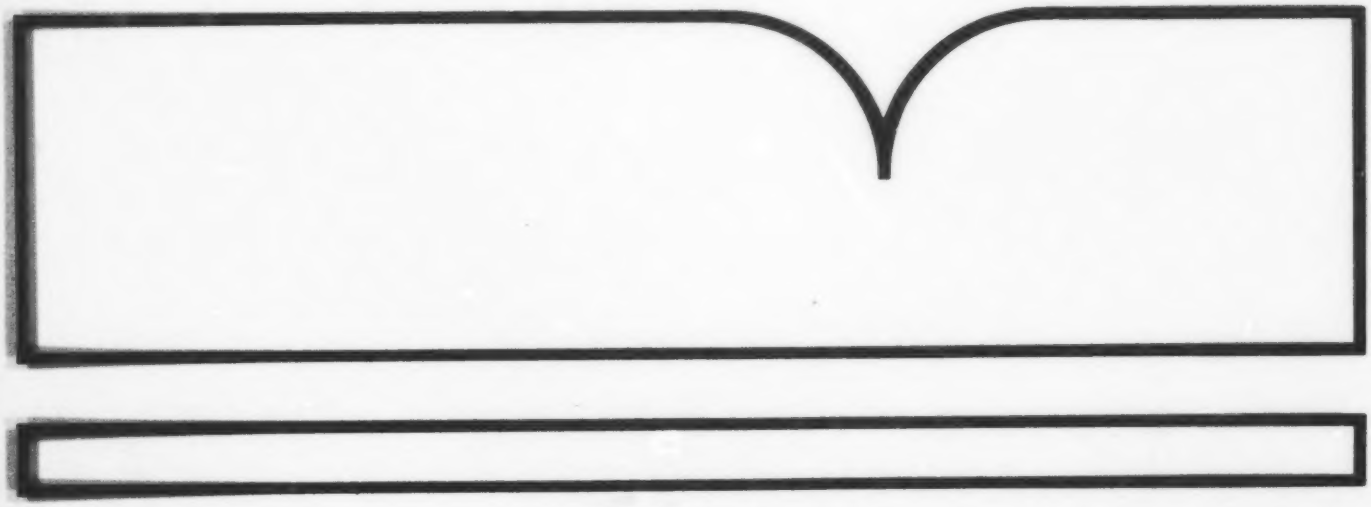

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CONVERSION OF MUNICIPAL SLUDGE TO OIL

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Produces more oil and energy and less by-product than secondary sludge

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ABSTRACT

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Thermal conversion of municipal sludge to oil has been investigated as a viable alternative for ultimate sludge disposal due to generation of energy. The conversion process using water as a solvent was evaluated in a batch mode using primary and secondary municipal sludges. A well defined oil separation procedure was developed in this research. The oil obtained after thermal conversion was dark brown, solid at room temperature and liquid at above 50°C, with an average heat content of 9400 cal/g oil. The increase of oil mass and energy yields as a result of thermal reaction was 73 and 67% in the primary sludge, and 515 and 485% in the secondary sludge, respectively, all with 10.9% volatile solids content. Catalyst addition did not increase the oil mass and energy yields. Data showed that primary sludge produces more oil, more energy, and less byproduct than secondary sludge in the thermal conversion process.

Keywords: Conversion, sludge, oil, energy, catalyst, separation.

CONVERSION OF MUNICIPAL SLUDGE TO OIL

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AND ATAL E. ERALP

INTRODUCTION

Sludge disposal has been a major challenge in the wastewater treatment area. Approximately half of the operating and maintenance costs of wastewater treatment plants are incurred by the sludge disposal processes. Current sludge disposal methods in the United States include incineration, landfilling, ocean disposal, and land spreading. All these methods have drawbacks; they are either too costly to operate or may create a potential threat to public health and the environment. Therefore, an economical sludge disposal process that ensures protection of public health and the environment needs to be developed.

Conversion of municipal sludge to oil has been investigated as a viable alternative for ultimate sludge disposal in recent years (1-4). This is because the technology not only eliminates sludge disposal problems, but also generates fuel energy in the form of oil that is transportable and marketable. Current conversion processes, all under development stages, may be classified into three major categories; pyrolysis (1,2), process using oil as a solvent (3), and process using water as a solvent (4). All of these processes are operated under an oxygen free environment.

Pyrolysis has been used in converting organics in municipal refuse and sludge to oil under high temperature, 450 to 800°C, and under pressure close to

atmospheric pressure(5). Recent progress in the pyrolysis process (1), however, employed low operating temperatures (250 to 320°C) with oil yields ranging from 20.2 to 27.4% from a feed sewage sludge of 40 to 50% total solids (TS). The reaction time was less than 2 hours including preheating time.

Young and Sachdev (2) evaluated a modified pyrolysis process (1) to demonstrate oil production from municipal sludge. Dried and finely ground feed sludge was subjected to thermal treatment in a 2.7 L Pyrex reactor for approximately 3 hours. The oil yields ranged from 12.8 to 28.7% based on TS (average is 20.4%), and energy content of oil ranged from 7600 to 9300 cal/g oil (average is 8636 cal/g oil). They reported that oil yield increased with temperature. However, the yield did not increase significantly above the optimum temperature. At 350°C, the oil yield was 12%, whereas at 400°C, the yield was 24.1%. They also reported that addition of catalysts such as Ni and Co did not increase oil yield. This was reasoned to be due to the complex nature of sludge that may already contain the necessary catalysts.

A process using oil as a solvent was operated under a hydrogen environment at operating pressures of 100 to 150 atm and temperatures of 300 to 425°C (3). Anthracene oil and sodium carbonate were added to completely dried and finely ground feed sludge as a carrier solvent and a catalyst, respectively. The oil yields ranged from less than 20% up to 50% at reaction times of 20 minutes or less. However, the investigator concluded that the process was not a viable one due to high cost of sludge drying, the complex nature of the process, and high ash content of the sludge.

The conversion process using water as a solvent was operated at a temperature of 275 to 350°C under a hydrogen (3) or a nitrogen (4) environment. The operating pressure was dictated mainly by water vapor pressure at a given operating temperature. Typically, the maximum operating pressure was less than 140 atm (2000 psi).

Kranich (3) used sewage sludge of approximately 5% TS for a batch operation in a 1 L stirred autoclave. The oil yields were 11 to 14% at 285°C and 27% at 295°C. Thermal reaction time was 15 to 20 minutes at 285 to 295°C, excluding preheating time. Apparent conversion of organics to oil was extremely variable and seemed not to depend on the addition of hydrogen or catalysts such as Ni and Mo. Most of the time, the oil and asphaltenes measured in the product were less than those in the feed sludge.

Molton et al. (4) produced feed sludge of 18.7 to 23.6% TS by centrifuging raw sludge with the aid of polymer. Sodium Carbonate was added as a reactant as 5% of wet feed sludge weight. Thermal reaction occurred in a continuous flow mode at a sludge feed rate of 720 L/d at operating temperature, pressure, and reaction time of 275°C, 150 atm, and 90 to 150 minutes, respectively. The oil yields ranged from 7.1% to 24.9% with an average of 15.3%. The oil content in the feed sludge ranged from 9.5 to 20.4% (average 15.9%). Heat content of both oils were 7678 to 8970 cal/g oil (average 8430 cal/g oil). Thus, the oil recovered in the product was slightly less than that present in the feed originally. This was the same as observed by Kranich (3) in a batch mode reaction.

Oil recovery from the reaction product appeared to be a major difficulty in the conversion process, although no researchers addressed the issue. The oil recovery less than the oil originally present in the feed may have been due to an inadequate oil recovery method. No clearly defined oil separation procedure has been reported, which is of major importance in practical application of the conversion process. Furthermore, no comparative study on oil yields from primary sludge and from secondary sludge (waste activated sludge) was conducted. Therefore, the objectives of this research were to determine:

1. Whether the process using water as a solvent can convert organics other than oily matter in sludge to oil.
2. The methodology of an oil separation procedure.
3. The effect of sludges with different oil and grease content and different origin, i.e. primary and secondary sludge, on the oil yield.

MATERIALS AND METHOD

A 1 L capacity stirred autoclave made of 316 stainless steel was used as an experimental reactor. The autoclave rated at 5000 psig at 345°C (650°F) is located at the U.S. EPA Test and Evaluation (T&E) Facility in Cincinnati, Ohio. Figure 1 shows the batch reactor system used in this study.

Primary and secondary sludges from the Little Miami Wastewater Treatment Plant in Cincinnati, Ohio was gravity thickened and/or centrifuged if higher solids content sludge was needed. Approximately 400 grams of wet sludge was used as feed sludge. When catalysts were added, the catalyst dosage was 5% of the wet weight of sludge. After adding feed sludge into a cold autoclave, nitrogen

gas was used to displace oxygen from the reactor and then to pressurize it to 100 psig. The initial nitrogen pressure was maintained as an indicator of pressure leakage. The electric heater surrounding the reactor's outer wall supplied needed heat to raise the reactor's temperature which took 1.5 hours. The operating temperature, pressure, reaction time, and stirrer speed were 345°C, 2500 psig, 1 hour, and 500 rpm, respectively. After completion of the reaction, the reactor was cooled down to room temperature which took 3.5 hours.

A reactor off-gas sample was collected in a Tedlar gas sampling bag. Reaction product was collected in a glass bottle using a 50 ml volume wide mouth pipet. The product still adhering to the reactor's walls and stirrer was recovered by dissolving it in Freon, and collected in a separate glass bottle. All glass sample bottles were cleaned with Freon.

A new separation method using Freon as an extraction solvent was developed in this study. The sample, whether reaction product or feed sludge, is adjusted to pH 2.0 using (1+1) HCL to convert fatty acid salts into free fatty acids. The free fatty acids are soluble in organic solvent; thus oil extraction from the sample can be maximized. As shown in Figure 2, approximately 50 grams of sample is added into a 200 ml separatory funnel and it's pH is adjusted to 2.0. Approximately 50 ml Freon is added into the separatory funnel and the mixture is shaken vigorously for 5 minutes. Freon becomes emulsified; however, spinning the funnel reseparates the Freon layer from the sample layer. The Freon layer containing oil is drained and fresh Freon is added again into the separatory funnel and the extraction procedure is repeated three more times.

Freon is evaporated from the Freon-oil mixture in a water bath of 65°C under open atmosphere. The end point of the evaporation was identified as the cessation of Freon bubble formation. Water from the water-solids mixture obtained from the separatory funnel is evaporated in a water bath of 100°C. Residue left after evaporation is dried in a drying oven of 103°C. Thus, the oil in this research is defined as Freon-dissolved substance for which the boiling point is greater than 65°C under open atmosphere and the char is defined as any substance after oil extraction and water evaporation.

Analytical parameters determined include total solids (TS), volatile solids (VS), Oil & Grease, total organic carbon (TOC), Heat of Combustion, and oil and off-gas compositions. Analytical methods for TS, VS, and Oil & Grease followed Standard Methods (6). TOC analysis used Astro's TOC Analyzer (7), and determination of Heat of Combustion used Parr Instrument's oxygen bomb calorimeter (8). Off-gas was analyzed using GC with flame ionization detector (6) and oil composition was determined using GC/MS with a capillary column (9).

RESULTS AND DISCUSSION

The oil from thermal reaction product was dark black, while that from feed sludge was light brown. Both oils are solid at room temperature and liquid at temperatures above 50°C, and are not volatile. The heat contents of both oils were similar, with an average of 9400 cal/g oil.

The oil yields from thermal reaction products and from feed sludge were measured using 2.65% VS (4% TS) primary sludge collected in July 1986 as feed

sludge to determine whether oil is produced from organics in feed sludge other than oily matter. Also, the effect of catalyst addition on the oil yield was investigated. Two different alkaline catalysts, Na_2CO_3 and NaOH, were tested with no catalyst addition as a control.

Figure 3 shows that yields of oil mass and energy increased when sludge was subjected to thermal reaction. The mass yields of oil defined as grams of oil obtained divided by grams of volatile solids of feed sludge were 21.8% from feed sludge, and 29.5% from reaction product. The energy yields of oil defined as calories in the oil obtained divided by calories in the sludge used were 35.5% from feed sludge, and 47.4% from reaction product. Relative increase in mass and energy yields of oil as a result of thermal reaction were 35% each. This indicates that oil is produced from organic matter in sludges other than oily matter.

Figure 3 also shows that mass and energy yields of oil among reaction products with different catalyst addition were similar. The average mass yields of oil were 30.9%, 29.7%, and 28.8% from reaction products without catalyst addition, Na_2CO_3 addition and NaOH addition, respectively. The average energy yields of oil were 49.4%, 47.1%, and 46.5%, respectively. This indicates that the addition of either Na_2CO_3 or NaOH as a catalyst into sludge in the conversion process does not increase oil mass and energy yields. All the oil yields data have been duplicated as a minimum to evaluate reproducibility. All data show relatively high reproducibility judging from less than 5% coefficient of variation of the oil yield data.

The analysis of oil composition by GC/MS in Figures 4 through 6 shows that two major peaks exist in each sample. Relative concentration and relative scan were defined as the ratio of mass of a compound to the total mass of all compound, and the ratio of scan of a compound to the total scan, respectively. Of these major compounds as shown in Table 1, Hexadecanoic acid was present in all reaction products and in the feed sludge with NaOH addition.

A comparison of oil composition from feed sludge and reaction product under no catalyst addition in Figure 4 shows that two major peaks are matching closely with similar relative concentration and scan. The second peaks in both oils is the same compound, Octadecanoic acid. Similar observation can be made in the case of sludge with NaOH addition. Two major peaks in both oils in Figure 5 are also matched closely with similar relative concentration and scan. The first peak is the same compound, Hexadecanoic acid. Besides these, small peaks in both oils in Figures 4 and 5 are quite similar. Thus, oils from feed sludge and thermally reacted reaction products under no catalyst addition as well as NaOH addition are similar in composition.

Oils obtained from Na_2CO_3 addition, however, exhibited different compositions. Although the relative scans of the two major peaks in both oils in Figure 6 are close together, the relative concentrations of the two peaks from the reaction product were almost twice as great as those from the feed sludge. The sum of the relative concentration of two peaks was .56 in the reaction product, while the sum was .30 in the feed sludge.

Table 1-Major compounds in oil from LMS (7/86).

ITEM	COMPOUND	RELATIVE CONCENTRATION	RELATIVE SCAN
SLUDGE (None)			
Feed	Unresolved mixture	.20	.57
	Octadecanoic Acid	.20	.64
Reaction	Hexadecanoic Acid	.23	.64
	Octadecanoic Acid	.19	.72
SLUDGE (NaOH)			
Feed	Hexadecanoic Acid	.15	.61
	Unknown	.11	.70
Reaction	Hexadecanoic Acid	.14	.66
	Unknown	.13	.74
SLUDGE (Na_2CO_3)			
Feed	Carboxylic Acid	.18	.61
	Decanoic Acid	.12	.69
Reaction	Hexadecanoic Acid	.30	.60
	Alcohol	.26	.68

The oil composition analysis shows similarity between oils from feed sludge and from reaction products as shown in Figures 4 and 5. However, differences exist in the case of Na_2CO_3 amended sludge as shown in Figure 6. This suggests that Na_2CO_3 may simplify oil composition, although a major portion of oil from thermal reaction product has been derived from oil already present in feed sludge. However, the catalyst addition does not increase the oil mass and energy yields. Some research reported that catalyst addition was not needed because sludge already contained numerous catalysts (5). Others (10) reported no increase of oil yields by adding Na_2CO_3 into Aspen wood which does not contain any catalysts. Youngquist et al (11) reported that addition of sodium carbonate to the sludge had no effect on yields and they attributed this to the presence of alkai salt in the sludge in sufficient quantities. Thus, no clear conclusion can be made as to the role and need of catalyst in the sludge-to-oil process.

The effect of different sources of sludge with different oil and grease content was evaluated using fresh batches of primary and secondary (waste activated) sludges from the Little Miami plant in September, 1986. Sludges were gravity thickened and centrifuged to simulate mechanically dewatered sludge. For comparison between two different sludges, the VS content of both sludges was adjusted to 10.9% based on wet sludge weight. The TS content of the primary and secondary sludges were 18.5 and 15.7%, respectively. The oil and grease content was 0.234 g per g of VS and 0.043 g per g of VS for primary and secondary sludges, respectively. Sodium carbonate was added as 25% of TS in the feed sludge.

Three separate thermal reactions and at least duplicate oil extractions from a reaction product were carried out for each sludge. Three separate oil extractions were also performed for feed sludges. Mass balances on solids were attempted

by measuring VS of all components in the oil separation steps including oil, char and sidestream water. Since oil is a desired product in the sludge-to-oil process, all mass and energy yields considered only mass and energy in the oil.

As shown in Table 2, the oil mass and energy yields from primary sludges were 0.182 g oil/g VS and 0.314 cal in oil/cal in feed under no thermal reaction conditions, and 0.313 g oil/g VS and 0.525 cal in oil/cal in feed under thermal reaction conditions, respectively. This corresponds to 73% and 67% increases in oil mass and energy yields, respectively, as a result of the thermal reaction. The yields from secondary sludge were 0.036 g oil/g VS and 0.066 cal in oil/cal in feed under no thermal reaction condition, and 0.221 g oil/g VS and 0.388 cal in oil/cal in feed under thermal reaction conditions, respectively. This corresponds to 515% and 485% increases in oil mass and energy yields, respectively, as a result of thermal reaction. These results clearly show that oil is produced from organic matter other than oil and grease originally present in feed sludge under thermal reaction conditions.

The GC/MS analyses of oils from primary and secondary sludges in Figures 7 and 8 show that two major peaks exist in three oil samples. The oil from the reaction product of the secondary sludge has several peaks of similar magnitude. As shown in Table 3 and Figures 7 and 8, the two major peaks are closely matched between feed and reaction product of the primary sludge. Considering the similar relative scans, the compounds in oil from the reaction product which were not specifically identified may well be Hexadecanoic acid and Octadecanoic acid.

Table 2 MASS AND ENERGY YIELDS OF OIL FROM LMS (9/86)

NO THERMAL REACTION

PRIMARY SLUDGE				SECONDARY SLUDGE			
RUN NO	Mo/Mf	Mo/Vf	Ho/Hf	RUN NO	Mo/Mf	Mo/Vf	Ho/Hf
PN1	0.1106	0.188	0.3254	SN1	0.0256	0.0373	0.0693
PN2	0.1108	0.1884	0.3279	SN2	0.0245	0.0357	0.0648
PN3	0.0993	0.1687	0.2903	SN3	0.0236	0.0344	0.0634
				SN4	0.0249	0.0362	0.066
AVG	0.1069	0.1817	0.3145				
STD DEV	0.0054	0.0092	0.0172	AVG	0.0247	0.0359	0.0659
				STD DEV	0.0007	0.0010	0.0022

THERMAL REACTION

PRIMARY SLUDGE				SECONDARY SLUDGE			
RUN NO	Mo*/Mf	Mo*/Vf	Ho*/Hf	RUN NO	Mo*/Mf	Mo*/Vf	Ho*/Hf
PR1A	0.1845	0.3135	0.5349	SR1A	0.153	0.2227	0.3961
PR1B	0.1898	0.3226	0.5514	SR1B	0.1581	0.23	0.4056
PR2A	0.1988	0.3379	0.5566	SR1C	0.1428	0.2079	0.3552
PR2B	0.2128	0.3616	0.6006	SR2A	0.1494	0.2173	0.3859
PR3A	0.1596	0.2712	0.4532	SR2B	0.1561	0.2271	0.4024
PR3B	0.1596	0.2713	0.4506	SR3A	0.1565	0.2277	0.4014
				SR3B	0.1499	0.2182	0.3878
AVG	0.1842	0.3130	0.5246	SR3C	0.1493	0.2172	0.3668
STD DEV	0.0195	0.0331	0.0551				
				AVG	0.1519	0.2210	0.3877
				STD DEV	0.0047	0.0068	0.0169

NOTE: Mo: mass of oil, gr.,
Ho: energy in the oil, cal,
Mf: TS in the feed, gr,
Vf: VS in the feed, gr,
Hf: energy in the feed, cal,
Mo*: (Mo x fa) + Mwo
fa: ratio of product recovered to product used for extraction,
Mwo: oil recovered from the reactor wall, gr,
Ho*: (Ho x fa) + Hwo,
Hwo: energy in the Mwo, cal.

Table 3

MAJOR COMPOUNDS IN OIL FROM LMS (9/86).

	COMPOUND	RELATIVE CONCENTRATION	RELATIVE SCAN
Primary sludge			
Feed	Hexadecanoic acid	.34	.45
	Octadecanoic acid	.22	.51
Reaction	Hydrocarbon or Alcohol	.41	.45
	Hydrocarbon or Alcohol	.24	.51
Secondary sludge			
Feed	Hexadecanoic acid	.36	.45
	Unknown	.24	.51
Reaction	Unknown	.11	.04
	Unknown	.11	.38
	Unknown	.11	.41
	Hydrocarbon or Alcohol	.19	.44
	Unknown	.15	.46

As shown in Figure 8, the composition of oil from the secondary sludge was quite simple compared to that of oil from the primary sludge as shown in Figure 7. The differences in oil composition may be due to consumption of a variety of easily biodegradable oil compounds in the influent wastewater by microorganisms during the microbial oxidation in the aeration basin. Apparent differences in oil composition between feed and reaction product of the secondary sludge cannot be explained.

Two major peaks in the oil from the feed secondary sludge match closely to those from the feed primary sludge. The unknown compound in the oil from the feed secondary sludge may well be Octadecanoic acid considering their similar relative scans. This suggests that both Hexadecanoic acid and Octadecanoic acid are resistant to microbial oxidation in the aeration basin, and they are major compounds of the oil component of the primary and secondary sludges.

A comparison of oil composition analyses between primary sludge of 4% TS (LMS 7/86) in Table 1 and Figure 4 and primary sludge of 18.5% TS (LMS 9/86) in Table 3 and Figure 7 reveals the same results. This was expected because different TS content of the feed sludges from the same source, although taken on different dates, would not affect the oil composition significantly. The close agreement between oil composition analysis results together with the small deviations among oil yields data strongly indicate that the experimental procedures developed in this study are highly reproducible and that analytical quality control was performed well.

VS mass balances were performed on sludges undergoing thermal reaction. As shown in Figure 9, fractional VS recoveries range from 0.66 to 0.80 (average

0.712). An incomplete VS recovery is due mainly to VS loss in the vent gas which is formed during the thermal reaction. The vent gas is of little interest in the sludge-to-oil process because it is largely a non-condensible gas; therefore, no quantitive analysis was made on the vent gas except its composition. It is composed mainly of CO_2 and nitrogen. Traces of oxygen were found, but no hydrogen was detected.

Although fractional VS recoveries are similar between reacted primary and secondary sludges, the contribution to the VS mass balance from different components was not the same. The VS contribution from oil, VS component of char and VS component of sidestream were 48.7, 16.9 and 34.4% of the reacted primary sludge VS and 30.4, 44.3 and 25.4% of the reacted secondary sludge VS, respectively. These results are due to differences in the feed sludge itself. Feed primary sludge contains a significant amount of oil (0.234 g oil/g VS) and VS components are composed of a variety of organic compounds. On the other hand, feed secondary sludge contains small amounts of oil (0.043 g oil/g VS) and VS components are composed largely of biological solids which are difficult to breakdown under the relatively low reaction temperature employed in this study. Therefore, primary sludge gives more oil per unit VS with less byproduct formation than secondary sludge.

As shown in Table 4, thermal reaction and alkaline pH increased the TOC content in the water phase (sidestream) obtained after oil separation. This was expected because of the vigorous nature of the thermal reaction. Also, chemical bonds of molecules can be destroyed more easily at alkaline pH than at neutral pH. Table 4 also showed that as the VS content of feed primary sludge increased

Table 4 - TOC in sidestream.

	Primary sludge		Secondary sludge	Primary sludge		Secondary sludge
VS, %	2.65	10.9	10.9	2.65	10.9	10.9
	mg TOC/g VS			mg TOC/L wet sludge		
No Reaction	46.7	13.1	26.8	1200	1430	2890
Reaction						
No Catalyst	110.1	-	-	2900	-	-
Na_2CO_3	128.3	54.0	100.8	3400	5900	10800
NaOH	182.6	-	-	4800	-	-

from 2.65% to 10.9%, TOC content in sidestream from thermal reaction products decreased from 128 to 54 mg TOC per g VS content of feed sludge. This is probably because of the relative increase of oil recovery in the product separation process. In other words, any product separation process is subject to an inherent oil recovery loss such as due to attachment to the walls of beakers and separatory funnels. Thus, by increasing VS content, more oil can be recovered per unit weight of VS applied. This results in a lower TOC content per unit of VS used in the sidestream. This observation indicates that an efficient recovery of oil during the separation procedure would reduce the sidestream strength.

TOC generation from secondary sludge was greater than that from primary sludge, both under conditions with and without thermal reaction. The TOC mass and concentration from secondary sludge was approximately twice that from the primary sludge. This may be due to the presence of excessive amounts of colloidal matter in secondary sludge compared to primary sludge, which results in a loss of organics during the oil separation procedure. Again, this indicates that an efficient oil recovery during the separation process would reduce the sidestream strength.

SUMMARY AND CONCLUSIONS

The feasibility of the conversion process from municipal sludge to oil using water as a solvent was evaluated. Emphasis was on data reproducibility and methodology rather than process optimization. A batch experiment was conducted using a 1 L stirred autoclave. Effects of thermal reaction, catalyst addition, and VS content of feed sludges on the oil yields were evaluated. The following conclusions were drawn from this research:

1. A well defined product separation procedure was developed.
2. Data showed conclusively that oil is produced by the conversion process from organics other than oily matter in the feed sludge.
3. The oil mass and energy yields were 0.313 g/g VS and 0.525 cal/cal in the primary sludge, and 0.221 g/g VS and 0.388 cal/cal in the secondary sludge, respectively, under thermal reaction conditions.
4. The increases of oil mass and energy yields as a result of thermal reaction were 73 and 67% in the primary sludge, and 515 and 485% in the secondary sludge, respectively.
5. Oil composition analysis indicates that majority of oil in the product may have been derived from oil already present in feed sludge.

6. Catalyst addition to feed sludge did not increase the oil mass and energy yields.
7. Primary sludge produces more oil, more energy, and less byproduct than secondary sludge in the conversion process.
8. Increases in VS content of feed sludge increased oil recovery and decreased TOC generation in sidestream. This is because of the relative increase in oil recovery as VS content increases.

REFERENCES

1. Bayer E. and Kutubuddin M., "Low temperature conversion of sludge and waste to oil". Proceedings of the International Recycling Congress, Berlin, West Germany (1982).

2. Young, D.N. and Sachdev, A.K., Liquid Fuel Production from Sewage Sludges, Dearborn Environmental Consulting Services, Ontario, Canada (1984).

3. Kranich, W.L., Conversion of sewage sludge to oil by hydroliquefaction. EPA Cooperative Agreement CR-807390010, EPA-600/S2-84-010; NTIS PB84-133768 WERL, Cincinnati, Ohio (1984).

4. Molton, P.M., et al. STORS: The sludge to oil reactor system. EPA cooperative Agreement CR-810690-01-0, WERL, Cincinnati, Ohio (1985).

5. EPA, Process Design Manual: Sludge Treatment and disposal, US EPA, EPA 625/1-79-011 MERL, Cincinnati, Ohio, (1979).

6. APHA, AWWA, WPCF, Standard Methods for Examination of Water and Wastewater. 16th ed., (1985).

7. Laboratory TOC Analyzer Operations Manual, Astro Model 1850, Astro Resources International Corporation, League City, Texas (77539).

8. Parr Instruments Instruction Manual, Parr Instrument, (1985).

9. Personal Communication (1986).

10. Eager, R.L., et al., ("Liquefaction of Aspen Poplar to Produce an Oil and Chemicals"). In Fundamentals of Thermochemical Biomass Conversion. Overend R. P. et al. (Eds.) Elsevier Applied Science Publishers (1985).

11. Youngquist, G.R. et al., "Conversion of Organic Wastes to Oil by Reductive Formylation". In Design and Management for Resources Recovery. Frankiewicz, T.C. (Ed) Ann Arbor Science, (1980).

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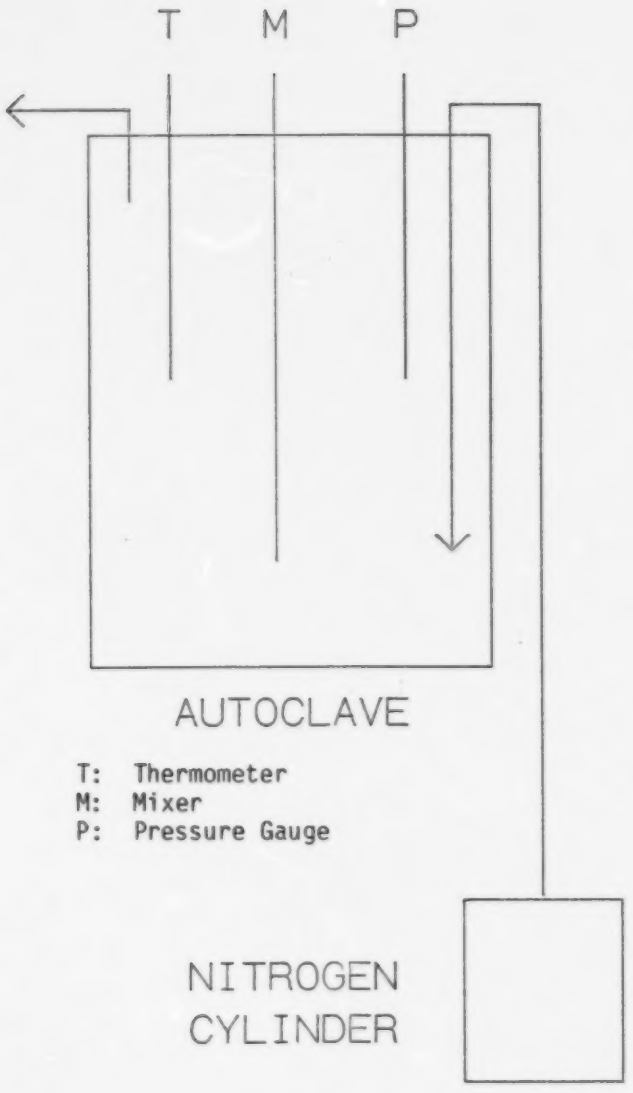

Figure 1. Schematic Diagram of the Reactor

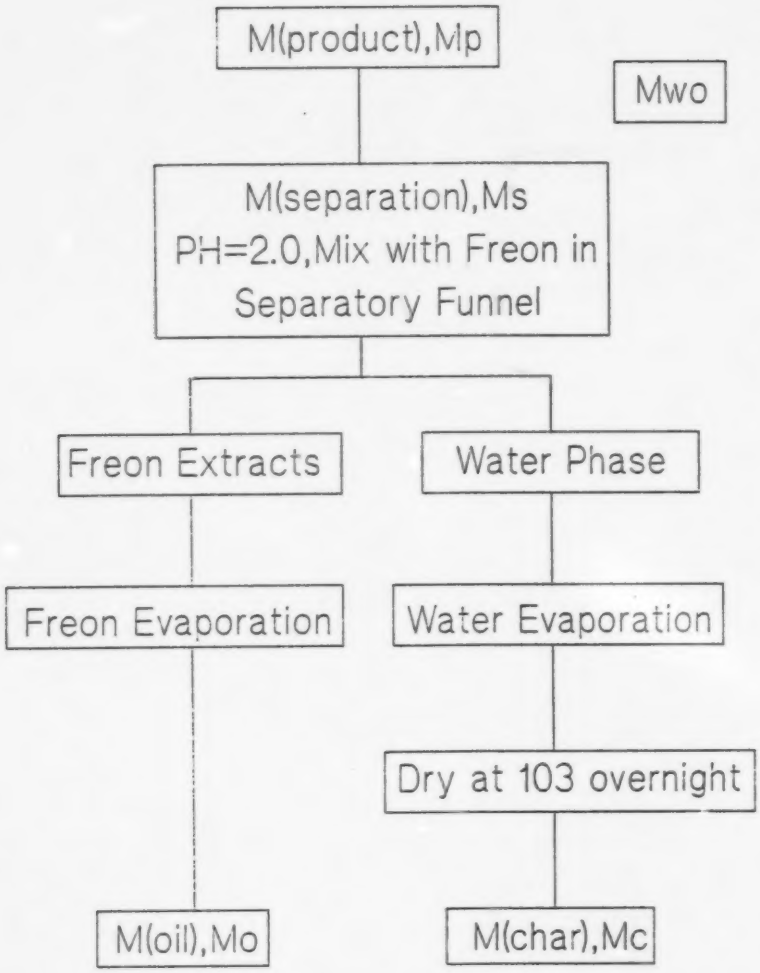

Figure 2. Product Separation Scheme

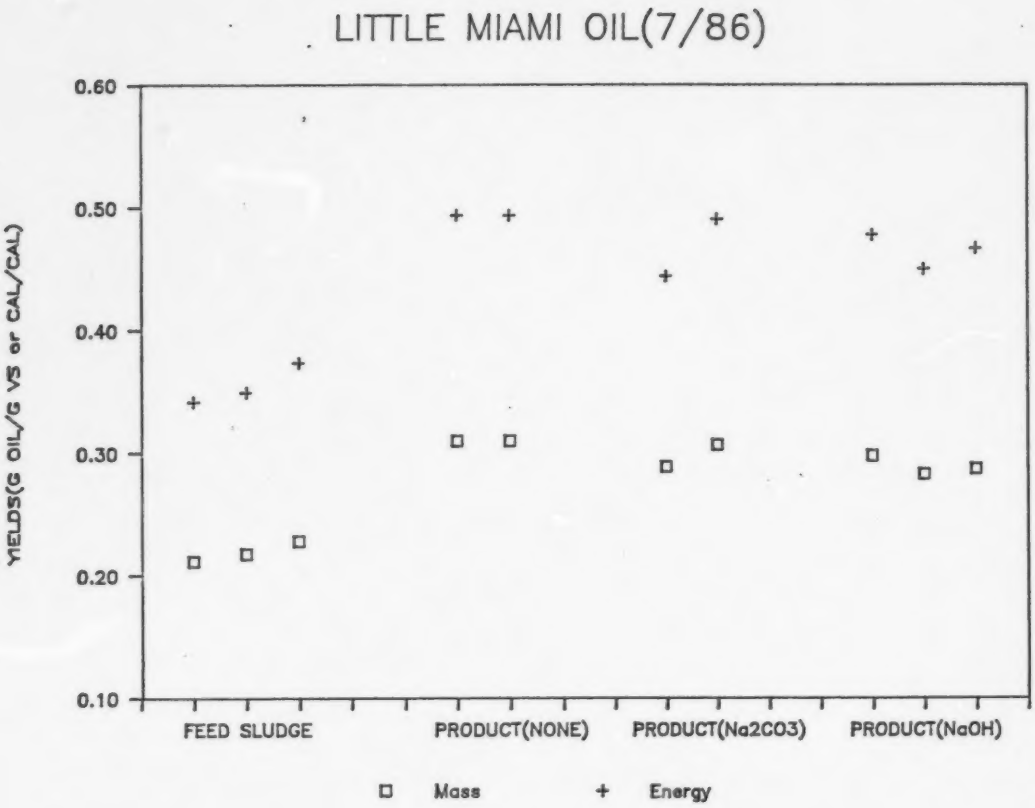

Figure 3. Mass and energy yields of Little Miami Sludge (7/86)

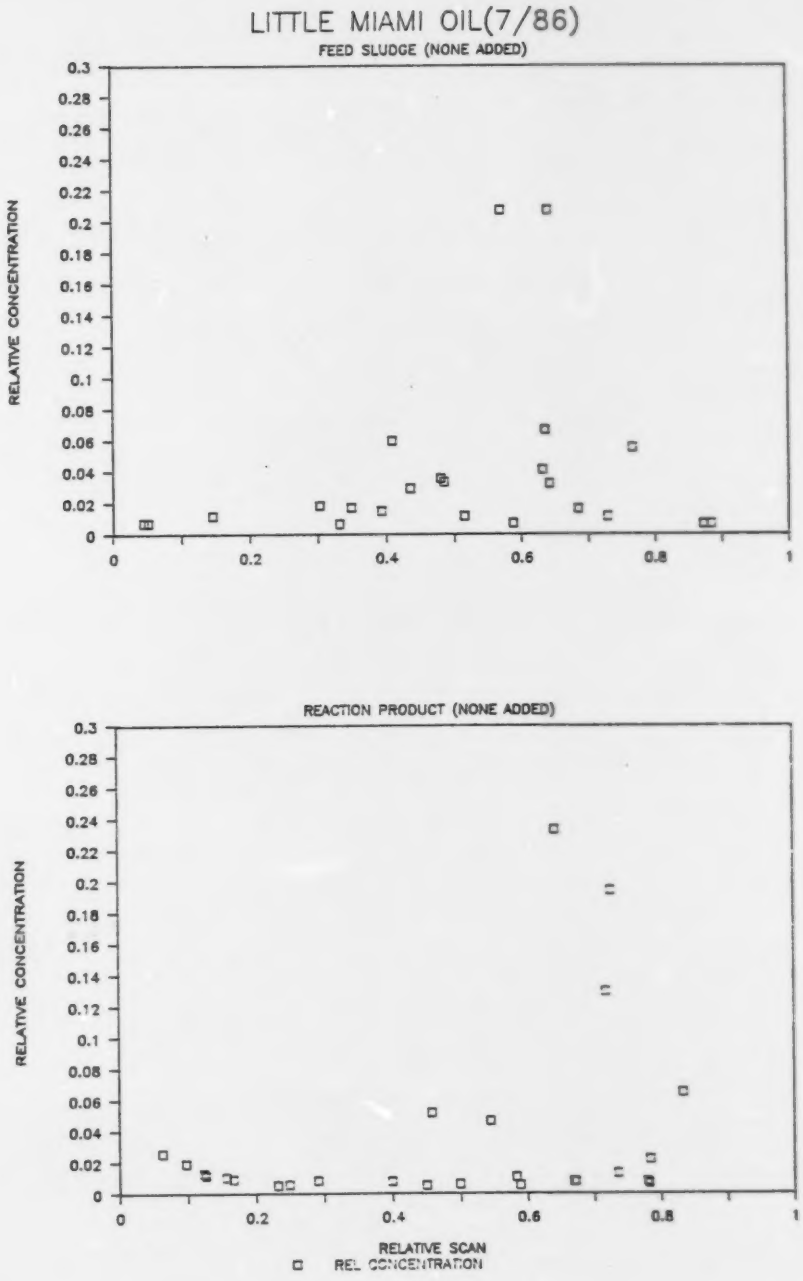

Figure 4. Oil composition of Little Miami sludge without catalyst additions

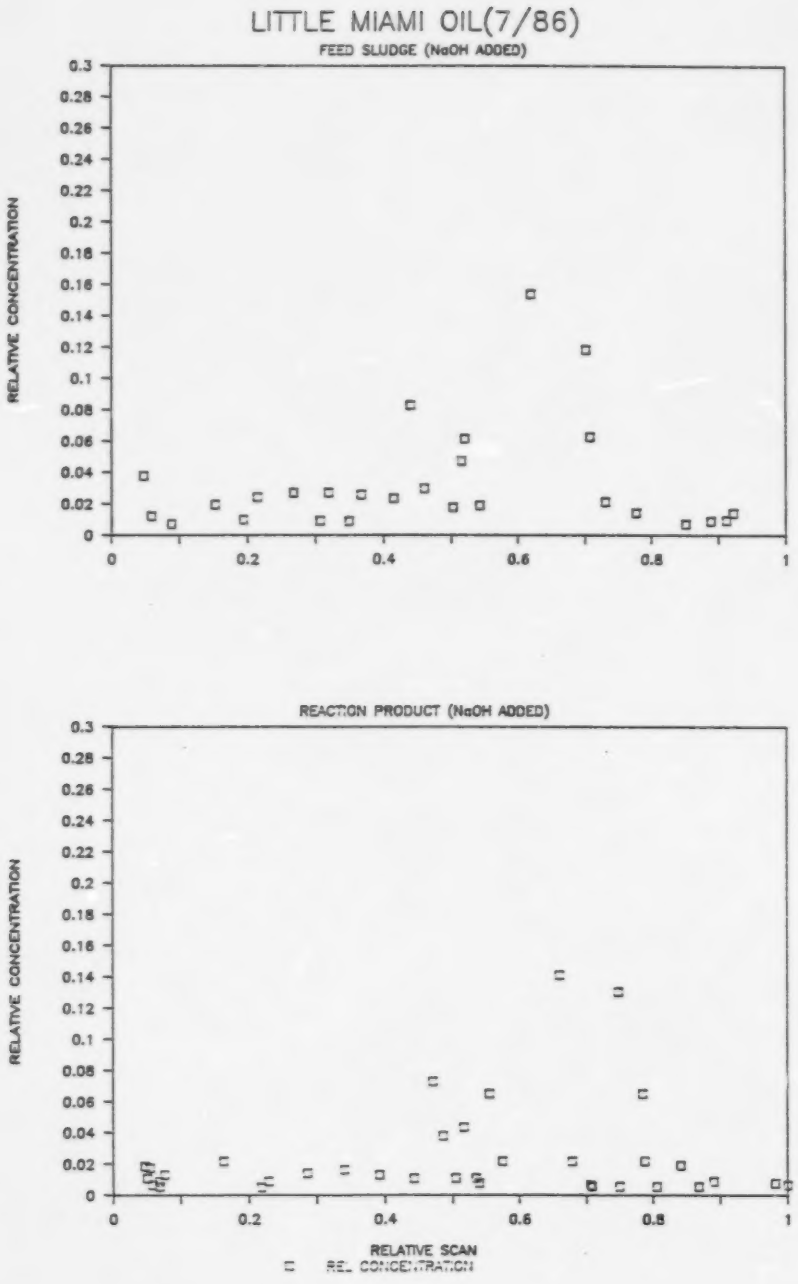

Figure 5. Oil composition of Little Miami sludge with NaOH additions

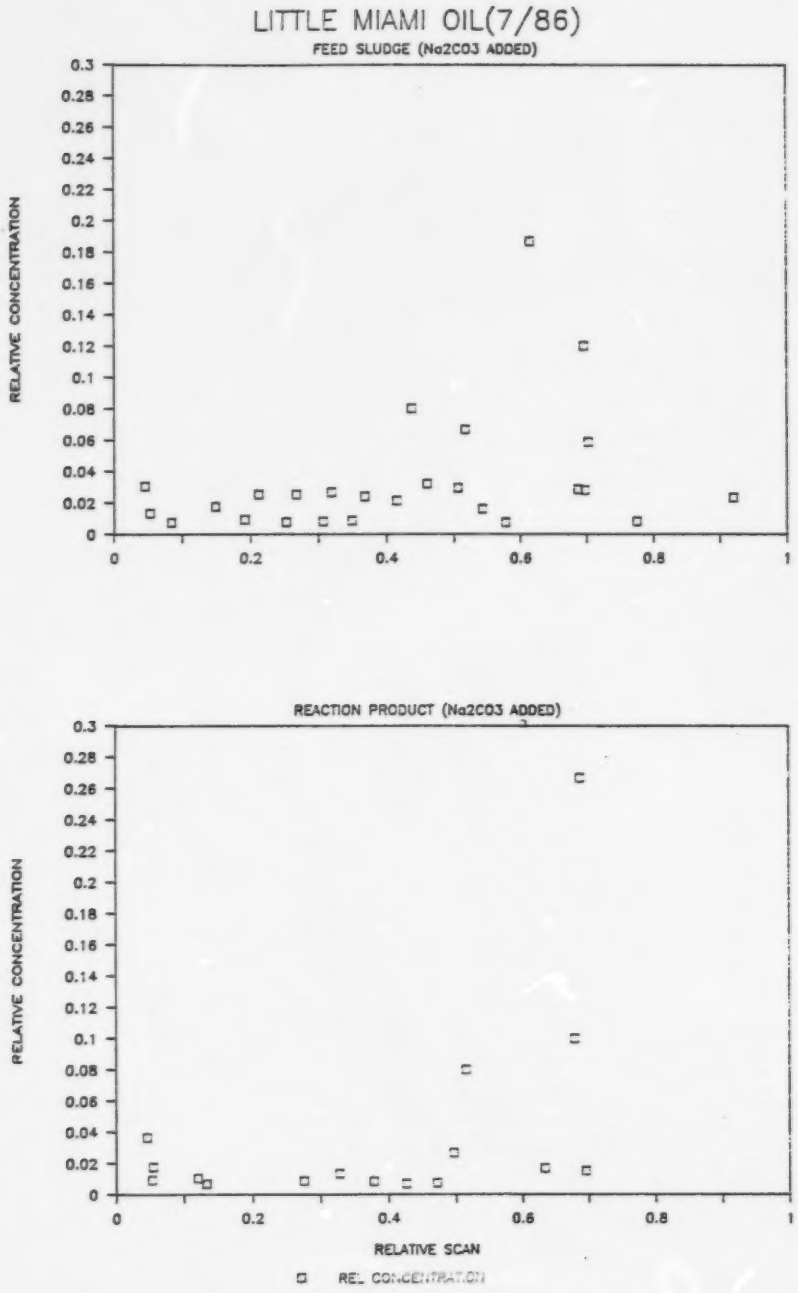

Figure 6. Oil composition of Little Miami sludge with Na_2CO_3 addition

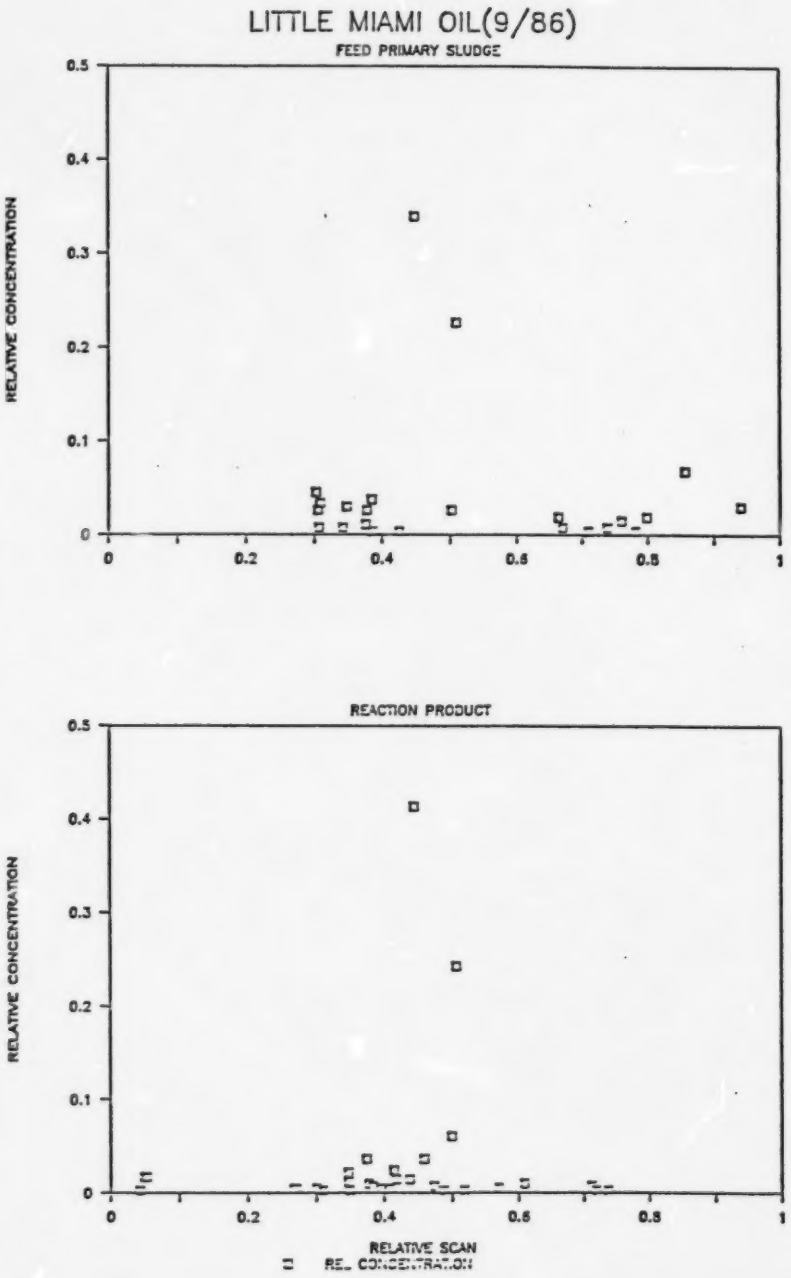

Figure 7. Oil composition of 10.9% VS primary sludge

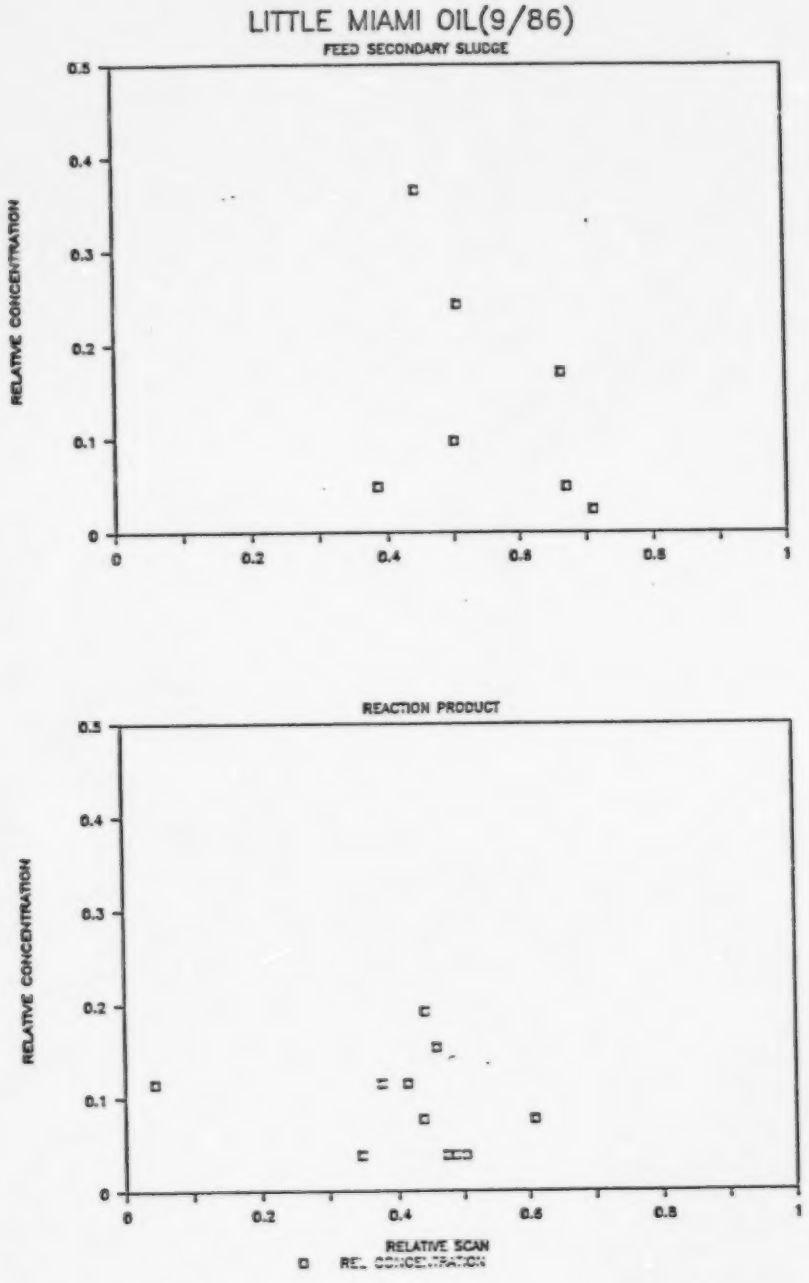

Figure 8. Oil composition of 10.9% VS Secondary Sludge

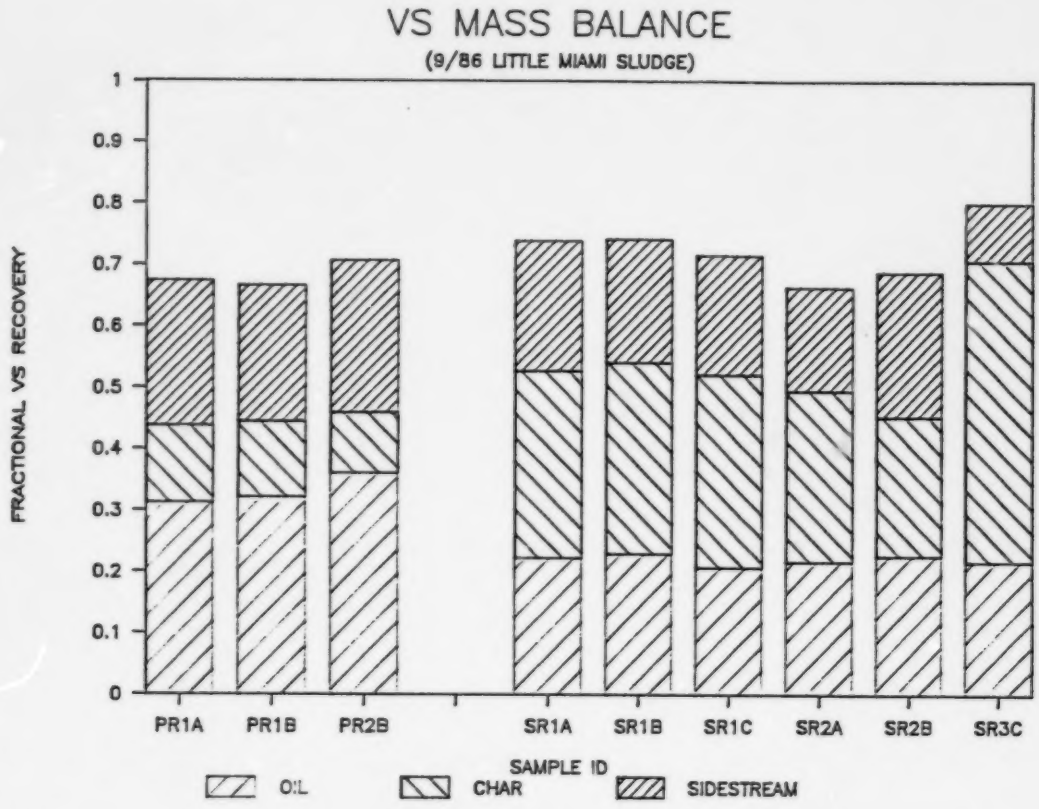

Figure 9. Fractional VS distribution among thermal reaction products